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EVALUATION OF THERMAL RESISTANCE IN CERAMICS

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The disadvantages of existing methods for determining the thermal resistance of materials are discussed. Based on data on the strength variation in samples of different compositions in thermal cycling, a non-labor-consuming and more objective method for evaluation of heat resistance is proposed. The testing conditions are substantiated.

The principles of thermal resistance of materials have been repeatedly refined, upgraded, and investigated and are set forth in sufficient detail in [1 – 4]. However, in spite of the large scope of theoretical and experimental studies, it is rather difficult to quantitatively assess the thermal stability on the basis of the formulas offered in the above studies. In practice, thermal stability is usually determined by using various experimental methods which to some extent take into account possible service conditions of materials and products under abrupt and disbalanced temperature variations. These approaches usually differ in the adopted method of thermal loading and the destruction criteria of tested samples used as the basis of the methods. The classification and the comparative analysis of numerous experimental methods for assessing thermal resistance are also widely discussed in the literature [5 – 7].

Without entering into a detailed critical analysis of the existent methods for determination of thermal resistance, let us point just to one significant disadvantage of all methods developed to test the resistance of materials to thermal loads, namely, the discrepancy between the experimental thermal loading conditions and the particular temperature and time service conditions of the material. Since the heat resistance parameters of different materials determined by various methods not only fail to correlate with one another but are often quite opposite, it is difficult to recommend a particular composite for long-term service under periodic and unbalanced temperature variations, using only the standard testing methods. The only possibility for a correct evaluation of the thermal stability of a product would be service-life testing under real conditions of variable thermal loads, in other words, it would be necessary to manufacture a product of a specific shape for each type of material, depending on its purpose and operation in terms of the service conditions of a thermoelectric set, up to the moment of failure permissible by the service conditions. However, such tests, on the one hand, would require substantial time, and on the other hand, cannot supply an answer on the degree of heat resistance of this ceramic under different service conditions.

Consequently, for a preliminary evaluation of the thermal resistance of materials it is desirable to approximate the testing parameters as close as possible to the actual service conditions and at the same time to confine the experiment duration to a realistic time frame. Thus, it is known that the stomatological furnace lining during a working day experiences the following thermal loads: heating for 1 – 1.5 h at a maximum rate up to the temperature of 1100 – 1500°C, then after a short holding, cyclic cooling to 400 – 500°C for 0.5 – 1 h, with subsequent heating again up to 1100 – 1150°C, and the number of these thermal cycles can be from 5 to 8. At a glance, the most expedient method for assessing the heat stability of developed lining materials can be thermal cycling up to the cracking or fracture of samples, i.e., the method commonly used to evaluate industrial refractories of granular structure [7, 8]. However, the number of thermal cycles for genuinely refractory materials (which quality is a necessary requirement on high-grade lining materials for stomatologic furnaces) can approach 1000 cycles or more (especially if cooling in performed in air), which makes the testing too labor-consuming.

Therefore, evaluation of the thermal resistance of lining materials used in stomatological or similar furnaces can, in our opinion, be based on the method of strength loss after several thermal cycles, which is sufficiently flexible and allows for obtaining ample data on the regularities of thermal destruction. As distinct from the standard method, instead of taking the percent strength loss after n thermal cycles as the thermal resistance parameter, in this case we suggest using the ultimate strength parameter, for instance, the sample bending strength after five heating-cooling cycles.

In fact, a higher level of strength loss after one thermal cycle does not even determine the inferior long-term service capacity of this material in terms of alternating heat loading (Fig. 1). Thus, samples of material 1 which after one cycle

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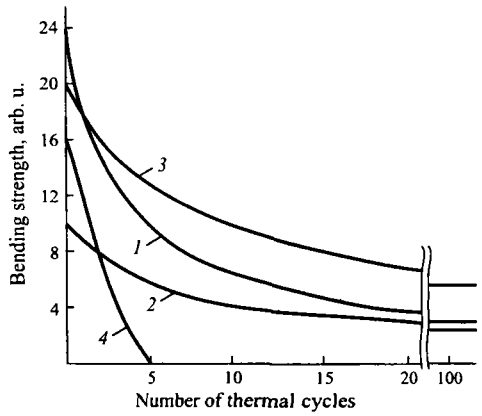

Fig. 1. Strength variations in different materials (*1* – *4*) in the course of thermal cycling.

lose 25% strength (from 24 to 18 arbitrary units) retain higher strength parameters during the overall long-term testing process than samples of material 2 which weaken only by 10% after one cycle. According to the standard evaluation method, the first material is less heat-resistant than the second one; however, its residual strength after one cyclic thermal shock stays higher (18 against 9) and, what is most important, the same tendency is retained in further testing. Therefore, the first material has to be regarded as the more heat-resistant with respect to service conditions and ability to withstand loads, including thermal loads.

Material 3 will undoubtedly be more heat-resistant compared to materials 1 and 2, and whereas with respect to the first material this conclusion does not depend on the evaluation method used (the strength loss after one thermal shock, or the residual strength after 5 thermal cycles), for the second material, this conclusion becomes evident only in using the recommended method, since according to the standard evaluation method, the thermal resistance of materials 2 and 3 appears to be equal (10% strength loss after a thermal shock).

It should be noted that the use of the phrases "thermal cycle," "thermal shift," and "thermal shock" in different methods is not always clearly defined. In fact, one thermal cycle or one thermal shock should provide for uniform slow heating of the tested samples up to a prescribed temperature, which excludes the emergence of stresses in the material, and subsequent rapid cooling in air or in water. Another version of a thermal cycle (thermal shock) is possible as well: the samples are placed in a heated furnace and then slowly (without stresses) cooled. Therefore, when the testing is implemented as the introduction of a sample into a heated furnace and then cooling in air or water, with subsequent repeating of these cycles, the correct term to use is "thermal cycle." The number of thermal shifts (thermal shocks) in this case will be twice as much as the number of thermal cycles. Unfortunately, in most cases researchers use these notions interchangeably, which hampers the comparative evaluation of heat resistance of different materials.

The number of thermal cycles after which the residual strength can be taken as the thermal resistance parameter is determined, on the one hand, by the wish to justifiably reduce the testing time frame to a certain minimum and, on the other hand, by the typical strength variations under thermal shocks. It is known that the most significant loss of strength occurs after initial thermal shocks, especially after the first one. Further decrease in strength, as a rule, is insignificant. Occasionally, it is even suggested that the thermal shock be used to increase thermal resistance, although the strength of the material in this case decreases (USSR patent 220815). In this context, five thermal cycles should be taken as the optimum number, after which a decrease in strength proceeds symbatically for all heat-resistant composites. If the weakening of samples is so abrupt that after five thermal shocks virtually no strength is registered (Fig. 1, material 4), such material can hardly be regarded as heat-resistant, although if the standard evaluation methods were used, its heat resistance would be comparable to the heat resistance of material 1 (the strength loss is 25% in both cases, i.e., from 16 to 12 arbitrary units).

The temperature of 800°C can be taken as the sample heating level in thermal cycling. This temperature is sufficiently high for developing substantial stresses in thermal cycling and, at the same time, in most ceramics this temperature does not yet cause plastic deformation facilitating the removal of thermal stresses through dissipation of a part of the thermal energy. Another important factor is that samples can be heated to the specified temperature level in the majority of laboratory furnaces. Samples in thermal cycling should be cooled in air or in water depending on the necessity of creating more or less severe thermal loads.

In accordance with the proposed method, the heat resistance of samples with different compositions produced in the development of ceramic materials for stomatologic furnace lining (RF patent 2116278) was evaluated. The strength variations in thermal cycling (800°C — water) of rod-shaped samples made of mixtures with varying ratios of G-00 alumina and refractory clay mostly were of a similar nature: all mixtures exhibited an abrupt decrease in strength after the first thermal cycle, and after five thermal cycles, the residual strength almost did not vary (Fig. 2). The only exception, which is of a rather probabilistic than regular nature, was registered in mixture with 40% alumina content. The experimental mixtures can be arranged in a consecutive series in order of decreasing residual strength after five thermal cycles, which relates to the increasing alumina content in the mixture. This regularity is preserved after 10 and 30 thermal cycles. At the same time, according to a heat resistance evaluation based on the strength loss after the first thermal cycle, mixture 3 is the most heat-resistant (14% loss), and mixture 4 is the least heat-resistant (38%); the other mixtures are approximately equal (32 – 33%)

In selecting the most heat-resistant mixture, the number of thermal cycles causing destruction was determined on solid cylindrical samples under rigorous thermal cycling conditions (800°C — water). The samples which contained 30% alumina withstood 241 cycles, those with 40% alumina withstood 236 cycles, 50% — 210 cycles, 60% — 180 cycles, and those with 70% withstood 143 cycles. Thus, the

same regularity was established as in determining heat resistance based on the residual strength after five thermal cycles.

These tests fully corroborate the lack of objectivity of the heat resistance evaluation method based on determining the loss of strength after a thermal shock and, at the same time, agree well with the proposed method for thermal resistance evaluation based on residual strength.

Another corroboration of the expediency of introducing a new parameter for assessing the heat resistance of ceramics was obtained on the basis of the strength variation registered in the thermal cycling (800°C – air) of the samples based on mixtures with different ratios of G-00 alumina and low-melting glass and fired at different temperatures (Table 1). The low values and the high dispersion in the determination of the average strength parameter of the samples with 20% glass content make the heat resistance evaluation based on strength loss unreliable. The heat stability of the samples containing 40% glass, judging from the strength loss, ought to be high. However, their strength parameters are significantly lower than the strength of the samples containing 60% glass in all stages of thermal cycling up to 75 cycles, which does not allow us to regard them as more heat-resistant. The equal initial strength and the similar nature of the strength variation in the thermal cycling of samples with 60% glass content gives grounds to speak of the equal thermal strength of the samples fired at different temperatures, in spite of the 4-fold difference in the strength loss values of some mixtures (1150 and 1225°C) compared to others (1300°C).

The situation becomes different if the bending strength of the samples after five (in this case, after seven) thermal cycles is taken as the heat resistance parameter. In this case, one can easily and objectively select the most heat-resistant compositions, correlate them, and assess the possibilities of their practical application.

Thus, evaluation of the thermal resistance of ceramic materials based on a parameter related to the bending strength after five heating-cooling cycles is free from of numerous disadvantages of the standard methods, closely simulates the actual service conditions, and makes it possible to obtain objective characteristics of the materials at minimum cost of the experiments. A thermal cycling involving 800°C — water or 800°C — air shifts can be recommended as the testing regime.

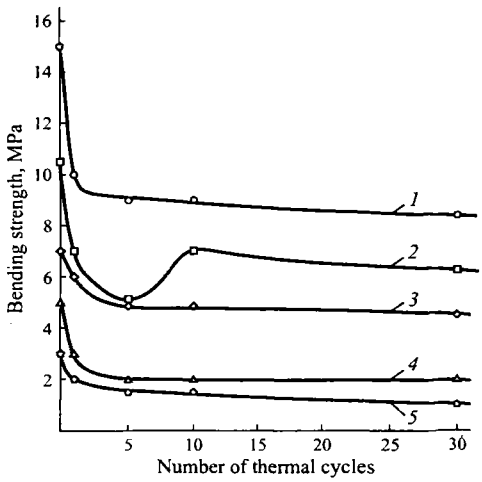

Fig. 2. Strength variations in samples in thermal cycling. Alumina content (%): *1*) 30; *2*) 40; *3*) 50; *4*) 60; *5*) 70; remainder is refractory clay.

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TABLE 1

Mixture	Glass content,* %	Firing temperature, °C	Bending strength, MPa, after cycles					Strength loss after 2 cycles, %
			0	2	7	25	75	
1	20	1150	0.7 ± 0.2	0.6 ± 0.1	0.8 ± 0.2	0.7 ± 0.2	0.3 ± 0.0	14
2	20	1225	1.0 ± 0.4	0.6 ± 0.4	0.4 ± 0.1	0.5 ± 0.1	0.3 ± 0.0	40
3	20	1300	2.0 ± 0.6	1.5 ± 0.2	1.4 ± 0.4	0.7 ± 0.2	0.4 ± 0.2	25
4	40	1150	7.4 ± 0.7	7.5 ± 0.4	7.0 ± 0.1	6.8 ± 1.3	5.4 ± 1.2	–1
5	40	1225	10.6 ± 0.8	10.7 ± 1.2	10.2 ± 1.4	10.3 ± 1.4	8.6 ± 1.6	–1
6	40	1300	10.5 ± 1.4	10.3 ± 0.6	9.9 ± 1.1	9.5 ± 3.4	7.7 ± 0.8	2
7	60	1150	20.9 ± 1.2	20.0 ± 1.1	18.0 ± 1.7	18.8 ± 1.3	16.8 ± 2.3	4
8	60	1225	21.3 ± 1.5	20.5 ± 0.4	21.2 ± 1.0	18.5 ± 1.8	18.6 ± 0.8	4
9	60	1300	20.5 ± 1.3	20.3 ± 1.0	19.0 ± 2.3	18.6 ± 1.1	18.2 ± 1.7	1

* Floor tile glaze.